

PATENT ABSTRACTS OF JAPAN

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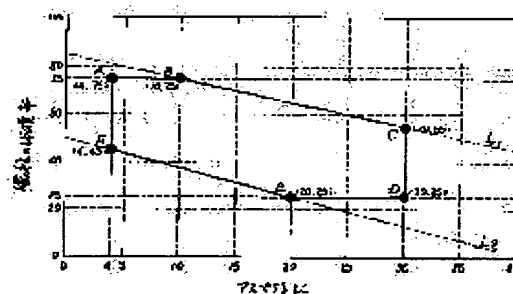
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(54) COMPOSITE MATERIAL AND ITS PRODUCTION

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a composite material reinforced by fibers composed of carbon and/or graphite and having low thermal expansion coefficient in facial direction and to provide a method for producing it.

SOLUTION: This composite material composed of carbon and/or graphite-series fibers and a matrix of copper or a copper alloy is the one composed of carbon and/or graphite-series fibers and a matrix of copper or a copper alloy, and, as for the carbon and/or graphite-series fibers, the length thereof exceeds $40\text{ }\mu\text{m}$, the aspect ratio and volume ratio lie in the range surrounded by the points A (4, 75), B (10, 75), C (30, 55), D (30, 25), E (20, 25) and F (4, 45) shown by the fig. 1, and, furthermore, they are oriented in the direction of the two-dimensional face at random. It has a low thermal expansion coefficient in the facial direction and is suitable as the material for a substrate for a semiconductor device.



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CLAIMS

[Claim(s)]

[Claim 1] It is constituted by the matrix which serves as carbon and/or fiber of graphite from copper or a copper alloy. Said carbon and/or the fiber of graphite A which the die length exceeds 40 micrometers and an aspect ratio and the rate of the volume show to drawing 1 (4 75), B (10 75), C (30 55), D (30 25), E (20 25), Composite material which consists of a matrix of the carbon which is within the limits surrounded by the point of F (4 45), and is characterized by making orientation carry out in the direction of a two-dimensional side at random and/or the fiber of graphite and copper, or a copper alloy.

[Claim 2] Composite material with which composite material consists of a matrix of the carbon according to claim 1 characterized by being used as a substrate for semiconductor devices and/or the fiber of graphite and copper, or a copper alloy.

[Claim 3] The manufacture approach of the composite material which loads the metal capsule of a bellows-like side attachment wall with the compression molding of the mixture which consists of powder of the fiber which consists of carbon and/or graphite, and a matrix, seals after degassing, and consists of a matrix of the carbon according to claim 1 or 2 characterize by make 1 shaft orientations contract the metal capsule which sealed said compression molding and/or the fiber of graphite and copper, or a copper alloy by hydrostatic-pressure pressurization between heat.

[Claim 4] The manufacture approach of composite material that the thickness for a covering device of a metal capsule consists of a matrix of the carbon according to claim 3 characterized by being more than twice with the thick side-attachment-wall section of a bellows-like metal capsule and/or the fiber of graphite and copper, or a copper alloy.

[Claim 5] The manufacture approach of the composite material which consists of a matrix of the carbon according to claim 3 characterized by loading the metal capsule of a bellows-like side attachment wall with compression molding, putting in a spacer, and sealing after degassing and/or the fiber of graphite and copper, or a copper alloy.

[Claim 6] The manufacture approach of the composite material which consists of a matrix of the carbon according to claim 3 to 5 with which compression molding of the mixture which consists of powder of the fiber which consists of carbon and/or graphite, and a matrix is characterized by being wrapped in copper sheet metal or a copper foil and/or the fiber of graphite and copper, or a copper alloy.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacture approach of composite-material ***** which starts the manufacture approach of composite-material ***** which consists of a matrix of carbon and/or the fiber of graphite and copper, or a copper alloy, especially consists of a matrix of carbon suitable as an ingredient of the substrate for semiconductor devices and/or the fiber of graphite and copper, or a copper alloy in the direction of a field with a low-thermal expansion coefficient.

[0002]

[Description of the Prior Art] The reinforcement and the metal which consist of a ceramic etc. are compound-ized, various ingredients which have both advantages are developed, and it is put in practical use widely. As one of them, the composite of carbon or graphite fiber, and copper is mentioned. This composite material is used for the collecting brush which harnessed the lubricity which carbon has, and the high electrical conductivity which copper has, the substrate for semiconductor devices of low-thermal expansion high temperature conductivity which harnessed the property of the low-thermal expansion which carbon or graphite fiber has, and the high temperature conductivity which copper has.

[0003] In case such a composite material is used as a substrate for semiconductor devices, the thing of a substrate which has arranged the carbon fiber artificially so that it is necessary to make low the coefficient of thermal expansion of the direction of a field at least, therefore thermal expansion may become low in the direction of a field is known (the conventional example 1, JP,59-16406,B, JP,58-16615,B).

[0004] Moreover, the aspect ratio of a carbon fiber mixes 200 or more comparatively long fiber, and solidifies, and what carried out orientation isotropic is known (the conventional example 2, JP,61-30013,B). Moreover, the radiator material for semiconductor devices obtained by mixing the short carbon fiber of 40 micrometers or less with the copper which is a matrix, or the powder of aluminum, and carrying out pressure sintering is proposed (the conventional example 3, JP,9-64254,A). Moreover, the thing which made the orientation of the carbon fiber carry out in the two-dimensional direction to the host phase of aluminum or aluminum alloy disorderly is proposed (the conventional example 4, JP,4-147654,A).

[0005] Moreover, as a process of composite material, powder-metallurgy processing is more common than before, hot pressing and the HIP method are often used for manufacture of composite material, such as metal powder and carbon, and the HIP method can especially be called approach excellent in mass-production nature. When manufacturing composite material by the HIP method, manufacture of composite material is made by sealing, after putting the compression-molding object of mixture into a metal capsule and deaerating it, and carrying out elevated-temperature high-pressure processing. When big deformation is performed by manufacture of the composite material by this HIP method, the idiosoma of a metal capsule and the core of a lid deform preferentially, and uneven deformation arises on the compression-molding object of a metal capsule. In shaping of the mixture of aluminum powder and reinforcement powder, using the side-attachment-wall section of a capsule as a bellows object is known about preventing uneven deformation of the capsule at the time of such HIP processing (the conventional example 5, the patent No. 2535401 official report).

[0006]

[Problem(s) to be Solved by the Invention] However, for arranging continuous glass fiber artificially in the above-mentioned conventional example 1, the problem that risk of local very big internal stress occurring if the hot heat history by Ag soldering etc. is added in what has arranged this continuous glass fiber, manufacture being unable to take time and effort, and a matrix being unable to bear [are hard to be good as economical, and] that internal stress, but breakage or deformation arising [an ingredient] is large is *****.

[0007] In the above-mentioned conventional example 2, fiber length is using 200 or more and comparatively long fiber by the aspect ratio. If hot pressing is used, the orientation of the fiber cannot be made to fully carry out in the direction of a field. And since it becomes that it is easy to be solidified, with fiber bent, very big internal stress will be accumulated into an ingredient, and it cannot be said that it is about dependability with it. [the large risk of breakage of the ingredient by the heat history, and deformation, and] [sufficient like the above-mentioned conventional example 1]

[0008] Moreover, in the above-mentioned conventional example 3, although the short fiber of 40 micrometers or less is used, very a lot of carbon fiber is needed for the coefficient-of-thermal-expansion fall effectiveness of each fiber becoming remarkably low, and obtaining a desired coefficient of thermal expansion, it is necessary to use the high high-class fiber of a degree of graphitization for obtaining high thermal conductivity in this case,

and, now, there is a problem of being high cost. Moreover, in the above-mentioned conventional example 4, although the orientation of the carbon fiber is made to carry out in the two-dimensional direction to the host phase of aluminum disorderly, when it applies to semiconductor devices, it cannot be said to be a thing with sufficient dependability.

[0009] moreover, although carry out the bellows object of the capsule side attachment wall section of HIP processing be show by the above-mentioned conventional example 5, this be a thing about shaping of the mixture of aluminum powder and reinforcement powder, and it be indicate about operation of arrange the orientation of the carbon in the matrix which consist of copper or a copper alloy, and/or graphite fiber at the same time it prevent the heterogeneous deformation of a capsule.

[0010] This invention is strengthened by the fiber which consists of carbon and/or graphite, has a low-feeve expansion coefficient in the direction of a field, and offers the manufacture approach of composite-material ***** which consists of a matrix of carbon suitable as an ingredient of the substrate for semiconductor devices and/or the fiber of graphite and copper, or a copper alloy especially.

[0011]

[Means for Solving the Problem] This invention is constituted by the matrix which serves as carbon and/or fiber of graphite from copper or a copper alloy. Said carbon and/or the fiber of graphite A which the die length exceeds 40 micrometers and an aspect ratio and the rate of the volume show to drawing 1 (4 75), B (10 75), C (30 55), D (30 25), E (20 25), It is within the limits surrounded by the point of F (4 45), and is the composite material which consists of a matrix of the carbon characterized by making orientation carry out in the direction of a two-dimensional side at random and/or the fiber of graphite and copper, or a copper alloy.

[0012] It is characterized by the composite material which consists of a matrix of the carbon of this invention and/or the fiber of graphite and copper, or a copper alloy being within the limits by which the aspect ratio and the rate of the volume were preferably surrounded by the point of H (5 70), I (15 60), J (20 60), K (20 40), L (10 40), and M (5 50) of drawing 2. In addition, in this invention, the rate of the volume is (volume of carbon and/or fiber of graphite)/(copper of the volume + matrix of carbon and/or the fiber of graphite, or volume of a copper alloy).

[0013] Moreover, composite material which consists of a matrix of the carbon of this invention and/or the fiber of graphite and copper, or a copper alloy is characterized by a matrix being copper or the copper alloy which contains one sort of Mo, W, Cr, Ag, and a ceramic particle, or two sorts or more further. Moreover, composite material which consists of a matrix of the carbon of this invention and/or the fiber of graphite and copper, or a copper alloy is characterized by being used as a substrate for semiconductor devices.

[0014] Moreover, this invention is the manufacture approach of the composite material which loads the metal capsule of a bellows-like side attachment wall with the compression molding of the mixture which consists of powder of the fiber which consists of carbon and/or graphite, and a matrix, seals after degassing, and consists of a matrix of the carbon characterize by to make 1 shaft orientations contract the metal capsule which sealed said compression molding and/or the fiber of graphite and copper, or a copper alloy by hydrostatic pressure pressurization between heat.

[0015] Moreover, the carbon of this invention, and/or the fiber and copper of graphite are also characterized by the thickness for a covering device of a metal capsule of the manufacture approach of composite material that ** consists of a matrix of a copper alloy being twice [thick / more than] the side-attachment-wall section of a bellows-like metal capsule. Moreover, the carbon of this invention, and/or the fiber and copper of graphite are also characterized by for the manufacture approach of composite material that ** consists of a matrix of a copper alloy loading the metal capsule of a bellows-like side attachment wall with compression molding, putting in a spacer, and sealing it after degassing.

[0016] Moreover, the carbon of this invention, and/or the fiber and copper of graphite are also characterized by wrapping the compression molding of the mixture which consists of powder of the fiber which the manufacture approach of composite material that ** consists of a matrix of a copper alloy becomes from carbon and/or graphite, and a matrix in copper sheet metal or a copper foil.

[0017] Furthermore, the manufacture approach of composite material that the carbon of this invention, and/or the fiber and copper of graphite also consist of a matrix of a copper alloy, as for ** is 800 degrees C or more, and hydrostatic-pressure pressurization between heat is characterized by being carried out at the temperature below the melting point of copper or a copper alloy.

[0018]

[Function] According to the composite material which consists of a matrix of the carbon of this invention and/or

the fiber of graphite and copper, or a copper alloy, it is strengthened by the fiber which consists of carbon and/or graphite, and has a low-thermal expansion coefficient in the direction of a field, and the internal stress by sintering solidification is small, and there is no risk of deformation by the heat history and breakage. Moreover, since it can consider as high temperature conduction in this direction by the coefficient of thermal expansion of the direction of a field perpendicular to a contraction shaft being able to take a low value, and using graphite fiber with the high rate of graphitization of high temperature conductivity, since carbon and/or the fiber of graphite are carrying out orientation of this invention in the direction of a two-dimensional side at random, if an ingredient is cut down to the parallel sense to this field, a product suitable as a substrate for semiconductor devices can be obtained.

[0019] Moreover, since 1 shaft orientations are made to contract manufacture of the composite material which consists of a matrix of the carbon of this invention and/or the fiber of graphite and copper, or a copper alloy using the metal capsule of a bellows-like side attachment wall by the hydrostatic-pressure pressurization between heat (the HIP method), the orientation of the fiber which consists of carbon and/or graphite can be made to carry out in the perpendicular direction to the contraction direction.

[0020] Since copper or a copper alloy is used for the composite material of this invention as a matrix, its thermal conductivity is good. Moreover, although the harmful carbide of aluminum Al_4C_3 grade is formed in the interface of the fiber of carbon and a graphite, and aluminum, it is easy to exfoliate in it in an interface at the time of a temperature cycle, and the heat expansion fall effectiveness by carbon and the graphite fiber is not fully acquired but a problem is in it in respect of dependability when a matrix is the composite material of aluminum, this problem cannot be found at the matrix of copper or a copper alloy. Furthermore, it has the thermal resistance in which Ag soldering with a package and a heat dissipation substrate is possible, and is useful also as a heat dissipation substrate to a ceramic package.

[0021] The aspect ratio of the carbon of this invention and/or graphite fiber and the reason for limitation of the rate of the volume are explained. Drawing 1 is drawing in which showing the rate of the volume of the fiber which becomes an aspect ratio and an axis of ordinate from carbon and/or graphite on an axis of abscissa, and showing the aspect ratio of composite material and the relation of the rate of the volume it is unrelated from the matrix of carbon and/or the fiber of graphite and copper, or a copper alloy. The aspect ratio and the rate of the volume of fiber which consist of the carbon and/or graphite of this invention are within the limits surrounded by the point of A (4 75), B (10 75), C (30 55), D (30 25), E (20 25), and F (4 45) shown in drawing 1.

[0022] Aspect ratios are fiber length/diameter of fiber. The fiber which the reason for limitation of the aspect ratio of this invention becomes from carbon and/or graphite with an aspect ratio smaller than 4 was difficult to produce, and since it became cost quantity, the four or more-aspect ratio thing was used for it. Moreover, the internal stress accumulated when the aspect ratio exceeded 30 and sintering solidification of the composite material is carried out became large too much, and since the risk of deformation by the heat history and breakage was large, it limited to 30 or less.

[0023] The rate of the volume of carbon and/or graphite fiber will become weak, even if sintering solidification becomes difficult and can sinter, if 75% is exceeded, the property as a composite material is not acquired, and the reason for limitation of the rate of the volume cannot fulfill the property especially demanded as a substrate for semiconductor devices. Moreover, since the rate of the volume becomes [a coefficient of thermal expansion] large too much and is not suitable if it is smaller than 25%, the rate of the volume is limited to 75% or less 25% or more.

[0024] Furthermore, in the field above the line L1 (alternate long and short dash line) in drawing 1, even if it fulfills these conditions, since the internal stress at the time of carrying out sintering solidification becomes large, and deformation and destruction are produced by the heat history, it is not suitable. Moreover, in a lower field, a coefficient of thermal expansion cannot be made more smaller than a line L2 (alternate long and short dash line). It cannot lower especially to the value of a coefficient of thermal expansion suitable as a substrate for semiconductor devices. The aspect ratio of carbon and/or graphite fiber and the rate of the volume of fiber were specified as the field surrounded by the point of A (4 75), B (10 75), C (30 55), D (30 25), E (20 25), and F (4 45) shown in drawing 1 for such a reason. Especially a desirable thing is the field surrounded by the point of H (5 70), I (15 60), J (20 60), K (20 40), L (10 40), and M (5 50) of drawing 2 which shows the relation between an aspect ratio and the rate of the volume.

[0025] Moreover, even if long fiber from which concentration of internal stress becomes a problem by that cause is not used for making the orientation of the fiber carry out in the direction of a two-dimensional side disorderly, it is because low-thermal expansion predetermined in the minimum amount of fiber specified at an aspect ratio and

the rate of the volume is obtained. Drawing 3 is drawing showing the outline of the composite material of this invention, and orientation of carbon and/or the graphite fiber (1) is carried out to the matrix (3) which composite material (2) becomes from copper or a copper alloy at random in the direction of a two-dimensional side.

[0026] A coefficient of thermal expansion with a composite material of this invention low in the direction of a field is obtained. When applying as a substrate for semiconductor devices, it is based on the gestalt of a semiconductor device, and its degree C is [degree C is suitable for a coefficient of thermal expansion in 5-11 ppm / ,] desirable 7.0-9.0 ppm /also in it, and the low-temperature expansion coefficient of the direction of a field of the composite material of this invention can respond to this demand.

[0027]

[Embodiment of the Invention] Although mentioned above about the aspect ratio and the rate of the volume of the carbon of this invention, and/or graphite fiber, a 5-20-micrometer thing is used and the path of carbon and/or the fiber of graphite is a 8-12-micrometer thing preferably. The die length exceeds 40 micrometers. In addition, the upper limit of the average die length of fiber becomes settled in the aspect ratio (fiber length/diameter of fiber).

[0028] Moreover, the matrix of this invention is copper or a copper alloy. as a matrix -- copper -- although it is good, the copper alloy which added one sort of Mo, W, Cr, Ag, and a ceramic particle or two sorts or more further may be used for copper or copper. Corresponding to an application, a strong rise is meant and one sort of refractory metals, such as Mo, W, and Cr, or two sorts or more are added. Moreover, a ceramic particle is added. Moreover, Ag etc. may be added in order to raise thermal resistance.

[0029] The manufacture approach of the composite material of this invention mixes the copper which makes a matrix for the fiber which consists of carbon or graphite by the technique of a ball mill etc. with the powder with which it is mainly concerned. Moreover, as a matrix, one sort of the powder of Mo, W, Cr, and Ag, two sorts or more, or a ceramic particle is added to Cu, the raw material mixture mixed and obtained is put in and pressed in a powder-compacting mold by the technique of a ball mill etc., and the compression molding of mixture is obtained.

[0030] the compression molding of the mixture which consists of powder of the fiber which consists of carbon and/or graphite, and a matrix -- HIP (hydrostatic-pressure pressurization between heat) -- the process which carries out sintering solidification using law is explained with reference to a drawing. As the process which carries out sintering solidification of the compression molding of this invention by HIP is shown and it is first shown in drawing 4 , the metal capsule which consists of [molding / compression / of carbon and/or the fiber (1) of graphite, and the powder (7) of a matrix / (8)] bellows objects (9) in a side attachment wall is loaded with drawing 4 - drawing 6 , and they carry out electron beam welding of the bellows object (9) to a lid (11) and (12) in a vacuum, and carry out vacuum seal.

[0031] As an ingredient of the metal capsule which consists of bellows objects (9) in a side attachment wall, stainless steel is mainly used. SUS304 is desirable from the point of weldability also in stainless steel. Moreover, as for a bellows object (9), and a lid (11) and (12), constituting from same ingredient is desirable. moreover, the metal capsule which consists of bellows objects in a side attachment wall -- the shape of cylindrical and an rectangular pipe -- any are sufficient. In respect of the yield at the time of the shape of a cylinder being desirable and obtaining a predetermined member from a billet in respect of the homogeneity of pressurization, the shape of an rectangular pipe is desirable. Moreover, although the bellows object of a metal capsule side attachment wall illustrated the radii-like thing, it may not be restricted to this and the bellows of 3 corniform is sufficient as it.

[0032] Moreover, if the rate of the volume of the fiber which consists of carbon or graphite becomes large, since the firmness of compression molding will be lost, it is desirable to wrap in copper sheet metal or a foil. For example, in accordance with the inside of the mold for powder compacting, it covers with copper foil (13) beforehand. By filling up with and carrying out powder compacting of the mixture of the fiber (1) which consists of carbon or graphite, and matrix fine particles (7) into it, as the compression molding (8) of a green compact takes the gestalt wrapped in copper foil (13), it gives firmness. As shown in drawing 5 , the metal capsule which consists of bellows objects (9) is loaded with a side attachment wall, a lid (11) and (12) are carried out, and it is desirable to carry out vacuum seal.

[0033] Subsequently, as shown in drawing 6 , the metal capsule of a bellows object (9) is loaded with a side attachment wall, and HIP processing of the compression molding (8) by which vacuum seal was carried out by the lid (11) and (12) is carried out. The bellows object (9) of a metal capsule side attachment wall is contracted to the shaft orientations of the fuselage. Although the reinforcement of the direction of a field perpendicular to

the shaft of a metal capsule fuselage will increase if contraction advances, contraction advances to shaft orientations further. As a result, in a field perpendicular to a shaft, anywhere, since the same pressurization contraction takes place, in this field, the volume ratio of the fiber (1) which consists of carbon or graphite, and orientation become the same. Thus, by pressurizing and shrinking 1 shaft orientations, the fiber (1) which consists of carbon or graphite becomes the thing which carries out orientation into a field perpendicular to contraction shaft orientations and by which orientation of carbon and/or the graphite fiber (1) is carried out at random to the matrix (3) in the direction of a two-dimensional side, and the composite material of this invention can obtain it by the easy and high yield.

[0034] Although orientation of carbon and/or the graphite fiber (1) is carried out to the matrix (3) at random in the direction of a two-dimensional side by pressurizing and shrinking 1 shaft orientations, drawing 7 (a) and (b) examined the composite of this invention about this. As shown in drawing 7 (a), a metal capsule (23) is loaded with the compression molding of carbon, the fiber (1) of graphite, and the powder (20) of a matrix, and vacuum seal is carried out by the lid (21) and (22). When HIP processing was performed to this, as shown in drawing 7 (b), the core of a metal capsule (23) and a lid (21), and (22) deformed preferentially, and, on the whole, the orientation of fiber (1) varied greatly according to the deformation situation.

[0035] Then, 1 shaft orientations are shrunk by the hydrostatic-pressure pressurization between heat (the HIP method), and the orientation of the fiber which consists of carbon and/or graphite is made to carry out in the perpendicular direction to the contraction direction in manufacture of the composite material of this invention using the metal capsule of a bellows-like side attachment wall. Moreover, even if it uses the metal capsule of a bellows-like side attachment wall, the ununiformity of capsule deformation may occur. For example, as shown in drawing 8, although the compression molding in which the metal capsule of a bellows object (9) was loaded with the side attachment wall, and vacuum seal was carried out by HIP processing of compression molding by the lid (17) and (18) is contracted to the shaft orientations of the fuselage which a side attachment wall becomes from a bellows object (9), the core of a lid (17) and (18) may deform and, on the whole, the orientation of fiber (1) may vary. Since it corresponds to such generating of heterogeneous deformation, by covering it the more than twice with a thick bellows object which makes a side attachment wall with the lid of a metal capsule, rather than a lid, the bellows object which forms a side attachment wall is made to transform preferentially, and pack density is fully raised. Since that a big pressure is applied to a lid can prevent until pack density fully goes up if it does in this way, the crater of a lid can be controlled.

[0036] A spacer may be used for the change which covers it twice [more than] as many thickness with a thick side-attachment-wall bellows object as this with the lid of a metal capsule. For example, as shown in drawing 9, the metal capsule of a side-attachment-wall bellows object (9) is loaded with the compression molding (8) wrapped in carbon and/or the fiber (1) of graphite, and copper foil (13) with the powder (7) of a matrix, a spacer (14) is put in, a lid (11) and (12) are carried out and vacuum seal is carried out. As the quality of the material of a spacer, oxygen free copper and a graphite are desirable. When HIP processing of this was carried out, as shown in drawing 10, it contracted to 1 shaft orientations and carbon and/or graphite fiber (1) carried out orientation of the side-attachment-wall bellows object (9) of a metal capsule to the matrix (3) at random in the direction of a two-dimensional side. Moreover, although the spacer inserted in a metal capsule is arranged on compression molding in drawing 9, you may arrange on compression molding and to the bottom.

[0037] The composite material which consists of a matrix of the carbon of this invention and/or the fiber of graphite and copper, or a copper alloy is explained with reference to drawing 11 about the case where it uses as a substrate for semiconductor devices. The composite material of this invention applies it to semiconductor devices, as orientation of carbon and/or the fiber of graphite is carried out in the direction of a two-dimensional side at random and fiber is carrying out orientation of such a composite material in the direction of a field of a semi-conductor substrate.

[0038] As specifically shown in drawing 11, it starts as fiber is carrying out orientation of the composite material of this invention in the direction of a field of a substrate, and a predetermined configuration is processed, and it uses as a substrate for semiconductor devices (4). The plating layer which becomes a substrate for semiconductor devices (4) from nickel, Au, Pd, etc. if needed is formed, and a semiconductor device (6) is carried on it. Moreover, the substrate for semiconductor devices (4) is joined to the envelope (5) of the package which consists of an alumina, covar, etc.

[0039] Thus, although the composite material of this invention is used as a substrate for semiconductor devices, it can consider as a suitable coefficient of thermal expansion to join to various semiconductor package ingredients by controlling an aspect ratio and the rate of the fiber volume. So, a terminal, curvature, etc. which

can control the curvature resulting from the differential thermal expansion at the time of joining to the envelope of the package which consists of this substrate, an alumina, covar, etc., and consist of a glass ceramic etc. can be joined, without generating. Moreover, since the thermal stress generated between the semiconductor chips generating heat can also be eased, a reliable semiconductor device is obtained.

[0040]

[Example 1] The 1st example of this invention is explained with reference to drawing 5, drawing 6 and Table 1, and Table 2. First, using 10 micrometers of diameters of fiber, graphite fiber of 200 micrometers of mean fiber length, and copper powder with a pitch diameter of 20 micrometers, weighing capacity of the fiber of predetermined weight and the copper powder was carried out, they were blended, it mixed with the ball mill, this was put into the metal mold which covered with copper foil with a thickness of 35 micrometers in accordance with the wall beforehand, the dry type press was carried out by the pressure of 1 t/cm², and compression molding was formed so that the rate of the volume of the fiber of graphite might become 50%. The filling factor (rate that the mixture of fiber and copper powder occupies among the volume of compression molding) of this compression molding was about 40%.

[0041] Subsequently, a metal capsule is loaded with the compression molding formed as mentioned above as shown in drawing 5. Vacuum enclosure was carried out by putting in into the metal capsule which has the bellows-like side attachment wall (9) with which the quality of the material consists of SUS304 the compression molding (8) of the copper powder (7) used as the graphite fiber (1) wrapped in copper foil (13), and a matrix by pitch 10.0mm of the bore of 65mm, the thickness of 0.6mm, and bellows, and carrying out electron beam welding of a lid (11) with a thickness of 2mm and (12) to the end face of this in a vacuum.

[0042] Subsequently, HIP processing was carried out on condition that 1500 atmospheric pressures, and 1000-degree-C2 h. As shown in drawing 6, the bellows-like side attachment wall (9) of a metal capsule was uniformly crushed in accordance with the shaft orientations of the cylinder by HIP processing, and compression molding was contracted by 1 shaft orientations. The thing with a height [of compression molding (8)] of about 60mm was contracted by 1 shaft orientations, and the fiber reinforced composite material with a height of about 25mm was obtained. Moreover, orientation of the graphite fiber (1) was carried out to the copper matrix (3) at random in the direction of a two-dimensional side.

[0043] As an example of a comparison, as shown in drawing 7 (a) and (b), HIP processing was performed. Like the above-mentioned example 1, compression molding was formed, the compression molding of the copper powder (20) used as graphite fiber (1) and a matrix was put into the metal capsule (23) of the shape of a cylinder with a bore [of 65mm], and a thickness of 0.6mm, and vacuum seal was similarly carried out by the lid (21) and (22). HIP processing of this was carried out on condition that 1500 atmospheric pressures, and 1000-degree-C2 h. After processing termination, when these were taken out from HIP equipment, as shown in drawing 7 (b), the amount of [of a metal capsule (23) and a lid (21) and (22)] core cratered greatly, it became a distorted form, and, on the whole, the orientation of fiber (1) varied greatly according to the deformation situation.

[0044] The with a diameter thickness [4mm thickness of 9mm] sample was extracted for every cm from the core about the composite material of the above-mentioned example 1 and the example of a comparison. In addition, a sample is extracted so that it may become the cylinder shaft orientations and the perpendicular of a metal capsule of HIP processing. Table 1 measures the consistency (g/cm³) of the sample of an example 1 and the example of a comparison, and Table 2 measures thermal conductivity (W/mK) with a laser flash method.

[Table 1]

密度(g/cm³)

中心からの距離(cm)	0	1	2	3
実施例	5.55	5.55	5.55	5.55
比較例	5.55	5.55	5.53	5.49

[Table 2]

熱伝導率(W/mK)

中心からの距離(cm)	0	1	2	3
実施例	390	387	392	389
比較例	391	381	365	339

[0045] A consistency is fixed at 5.55 (g/cm³), and all whose distance from a core a sample extraction location is 0cm, 1cm, 2cm, and 3cm in the example 1 so that clearly from Table 1 are **. On the other hand, in the example

of a comparison, dispersion has arisen in the consistency. Moreover, the thermal conductivity shown in Table 2 is not called at a sample extraction location in the example 1, either, but is almost fixed. On the other hand, dispersion has produced the example of a comparison.

[0046] From the result of these tables 1 and Table 2, in the example 1, it does not call at a sample extraction location, but since the consistency is fixed, it can be said that the volume ratio of fiber and the copper of a matrix is fixed. Moreover, since thermal conductivity is fixed, it does not call at the extraction location of a sample, but it can be said in the sense perpendicular to a compression shaft that the orientation condition of fiber is the same anywhere. To it, in the example of a comparison, dispersion arose also in the consistency and further much dispersion arose in thermal conductivity. As shown in drawing 7 (b) from this, distorted deformation of a capsule shows that the volume ratio of fiber and orientation change greatly with sample extraction locations.

[0047] It was 10.2, when the sample of this example 1 was dissolved using the acid and having been asked for the average aspect ratio. In addition, although the graphite fiber of a raw material was 10 micrometers of diameters of fiber, and the thing of 200 micrometers of mean fiber length, the average aspect ratio of the composite material obtained by mixing with a ball mill, compression molding by the dry type press, or HIP was the thing of 10.2.

[0048]

[Example 2] The 2nd example is explained with reference to drawing 12 (a), (b), and drawing 13 (c) and (d). In drawing 12 (a), (b), and drawing 13 (c) and (d), an axis of abscissa is a rate of the fiber volume which an aspect ratio and an axis of ordinate become from carbon and/or graphite. 10 micrometers of diameters of fiber and the composite material which has various aspect ratios and various rates of the volume by the same process as an example 1 by making a Cu-20vol%Mo alloy into a matrix were produced as fiber of carbonaceous. This was heat-treated 2h at 850 degrees C among reducing atmosphere, and the deformation behavior was investigated. Moreover, the coefficient of thermal expansion was measured and it investigated whether it was a suitable value (5-11 ppm). It investigated whether these would be synthesized and each composite material would be equipped with the property suitable as a substrate for semiconductor devices.

[0049] Drawing 12 (a) shows the produced composite material by O mark. what drawing 12 (b) was what showed the deformation behavior by heat treatment, O mark had the thing not deforming or slight deformation, and x mark transformed remarkably -- or it destroys. It is what showed the deformation behavior by heat expansion, and the thing of a value (5-11 ppm) with appropriate O mark and x mark are too high, or drawing 13's (c)'s are too low. Drawing 13 (d) is what showed the comprehensive evaluation result, and its thing and x mark for which O mark is suitable as a substrate for semiconductor devices are unsuitable as a substrate for semiconductor devices. Only when it has the aspect ratio and the rate of the volume within the limits which are specified by this invention so that clearly from the result shown in drawing 12 (a), (b), and drawing 13 (c) and (d), it turns out that it has a suitable property.

[0050]

[Example 3] The 3rd example is explained with reference to drawing 11 . With 10 micrometers of diameters of fiber, and a pitch diameter [the fiber of the graphite of 200 micrometers of mean fiber length and the pitch diameter of 10 micrometers] copper powder was blended so that the rate of the volume of graphite fiber might become 60%, and composite material was produced like the example 1. It was 7.5 when asked for the aspect ratio like the example 1. This composite material was cut down so that the orientation side of graphite fiber might serve as the direction of a field, it considered as the substrate, and as shown in drawing 11 , Ag soldering of the envelope (5) which becomes a substrate for semiconductor devices (4) from an alumina was carried out. Under the present circumstances, curvature etc. was not looked at by the substrate (4). Furthermore, a semiconductor device (6) is attached in this and they are 500 cycles or a beam about a -40 degree-C<-->125 degree C temperature cycle. Consequently, the exfoliation from the substrate (4) of a semiconductor device (6) was not accepted.

[0051]

[Example 4] The 4th example is explained with reference to drawing 5 - drawing 10 and Table 3, and Table 4. Raw material mixture was mixed like the example 1 mentioned above, compression molding was formed, and the same HIP processing as an example 1 was carried out. As shown in ("the lid thickness of 2.0mm" of Table 3 and Table 4), and drawing 5 , one put compression molding into the metal capsule by which a side attachment wall consists of bore [of 150mm], thickness [of 1.0mm], and pitch 15mm bellows **** (9), and it carried out the lid (11) whose thickness is 2.0mm, and (12), and carried out vacuum enclosure.

[0052] As shown in (the "lid thickness [of 0.6mm] + spacer" of Table 3 and Table 4), and drawing 9 , another loaded with compression molding the metal capsule by which a side attachment wall consists of bore [of 150mm], thickness [of 1.0mm, and pitch 15mm bellows **** (9), put in the spacer (14) with a thickness of 10mm it is thin from oxygen free copper, carried out the lid (11) whose thickness is 0.6mm, and (12), and carried out vacuum enclosure Furthermore, ("lid thickness of 0.6mm" of Table 3 and Table 4), as shown in drawing 8 , compression molding was put into the metal capsule by which a side attachment wall consists of bore [of 150mm], thickness [of 1.0mm], and pitch 15mm bellows **** (9), and thickness carried out the lid (17) which is 0.6mm, and (18), and carried out vacuum enclosure. In addition, as for the metal capsule of a bellows object, and the quality of the material of a lid, all and a case also used SUS304.

[0053] As a result of performing HIP processing, as shown in drawing 6 in the case of "2.0mm lid thickness" shown in drawing 5 , it is mutually parallel, and the crater etc. was not seen, but a lid (11) and (12) contracted compression molding to 1 shaft orientations. Moreover, although the thickness of a lid was 0.6mm, since the spacer with a thickness of 10mm was put in in the case of the "lid thickness [of 0.6mm] + spacer" shown in drawing 9 , the crater etc. was not generated as shown in drawing 10 . On the other hand, by "the lid thickness of 0.6mm", as shown in drawing 8 , the core of a lid (17) and (18) deformed. As for the deformation, the center section of a lid (17) and (18) had caved in about 3mm to the periphery.

[0054] About these ingredients, the sample was extracted like the above-mentioned example 1, and a consistency and thermal conductivity were measured. Table 3 shows a consistency (g/cm³) and Table 4 shows thermal conductivity (W/mK).

[Table 3]

密度(g/cm³)

中心からの距離(cm)	0	1	2	3
蓋厚み2.0mm	5.55	5.55	5.55	5.55
蓋厚み0.6mm + スペース	5.55	5.55	5.55	5.55
蓋厚み0.6mm	5.55	5.55	5.54	5.52

[Table 4]

熱伝導率(W/mK)

中心からの距離(cm)	0	1	2	3
蓋厚み2.0mm	390	387	392	389
蓋厚み0.6mm + スペース	392	389	387	390
蓋厚み0.6mm	391	388	378	365

[0055] "The lid thickness of 2.0mm" and a "lid thickness [of 0.6mm] + spacer" cannot call at a sample extraction location, but from the result of Table 3 and Table 4, since the consistency is fixed, the volume ratio of fiber and the copper of a matrix is fixed, and with the sense perpendicular to a compression shaft, the orientation condition of fiber is the same anywhere, and it can be said that thermal conductivity is fixed. To these, in what the crater with a "lid thickness of 0.6mm" generated, dispersion arose also in the consistency and dispersion arose also in thermal conductivity.

[0056]

[Effect of the Invention] As explained above, according to the composite material and its manufacture approach of this invention, the effectiveness that it is strengthened by the fiber which consists of carbon and/or graphite, and has a low-ferve expansion coefficient in the direction of a field, and the internal stress by sintering solidification is small, and there is no risk of deformation by the heat history and breakage is done so.

[Translation done.]

* NOTICES *

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1.This document has been translated by computer. So the translation may not reflect the original precisely.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] Drawing showing the relation between the aspect ratio of this invention, and the rate of the volume

[Drawing 2] Drawing showing the relation between the aspect ratio of this invention, and the rate of the volume

[Drawing 3] Drawing showing the outline of the composite material of this invention

[Drawing 4] Drawing explaining the embodiment and example of this invention

[Drawing 5] Drawing explaining the embodiment and example of this invention

[Drawing 6] Drawing explaining the embodiment and example of this invention

[Drawing 7] Drawing showing the example of a comparison

[Drawing 8] Drawing showing the example of a comparison

[Drawing 9] Drawing explaining the embodiment and example of this invention

[Drawing 10] Drawing explaining the embodiment and example of this invention

[Drawing 11] Drawing explaining the embodiment and example of this invention

[Drawing 12] Drawing showing the relation between the aspect ratio of the example of this invention, and the rate of the volume

[Drawing 13] Drawing showing the relation between the aspect ratio of the example of this invention, and the rate of the volume

[Description of Notations]

1 Fiber Which Consists of Carbon or Graphite

2 Composite Material

3 Matrix

4 Substrate for Semiconductor Devices

5 Envelope of Semiconductor Device

6 Semiconductor Device

7 Powder of Matrix

8 Compression Molding

9 Bellows Object

11 12 Lid

13 Copper Foil

14 Spacer

[Translation done.]

* NOTICES *

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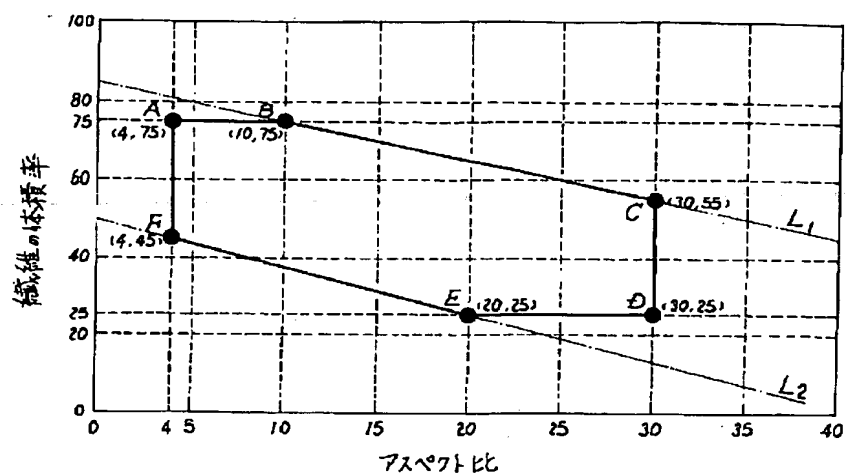
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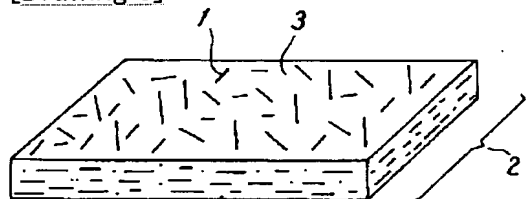
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DRAWINGS

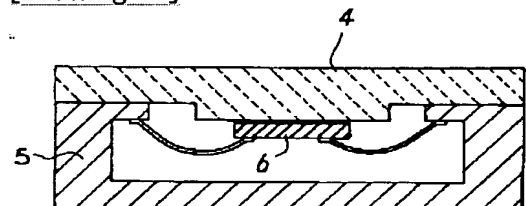
[Drawing 1]



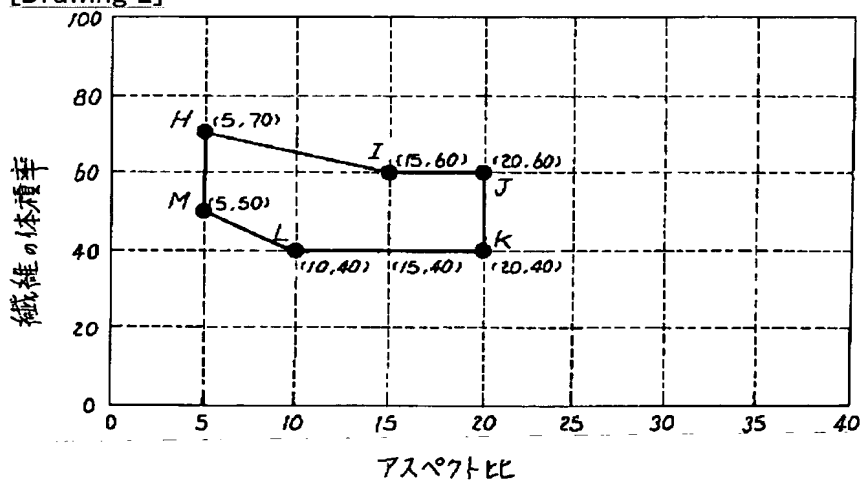
[Drawing 3]



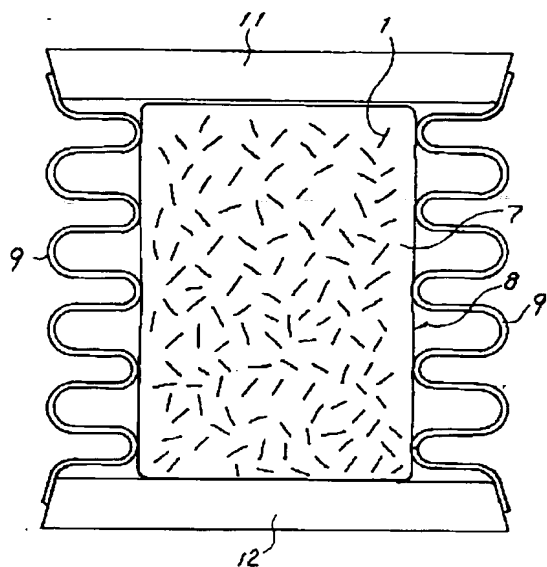
[Drawing 11]



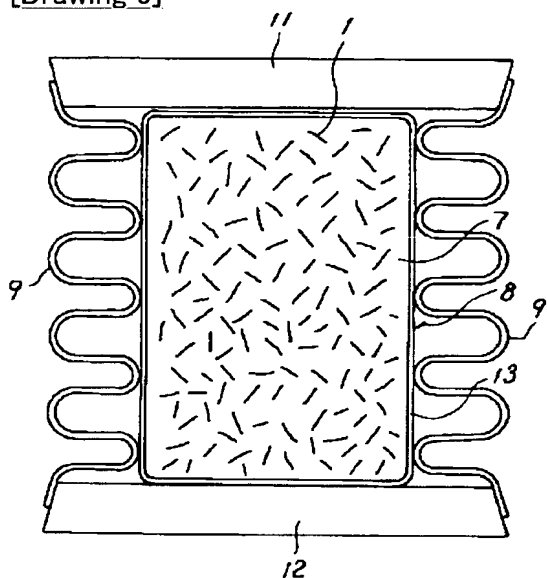
[Drawing 2]



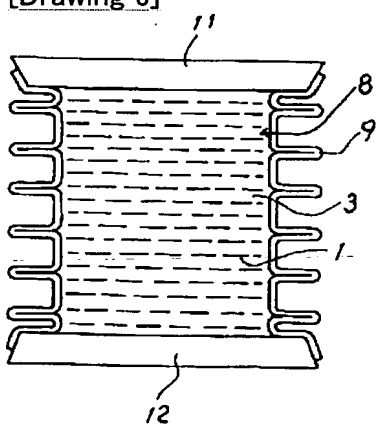
[Drawing 4]



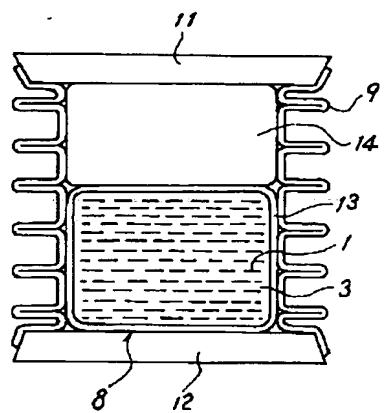
[Drawing 5]



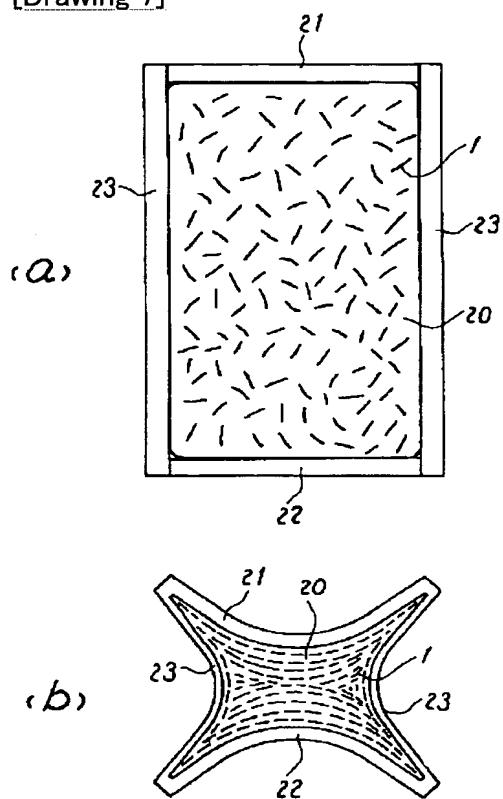
[Drawing 6]



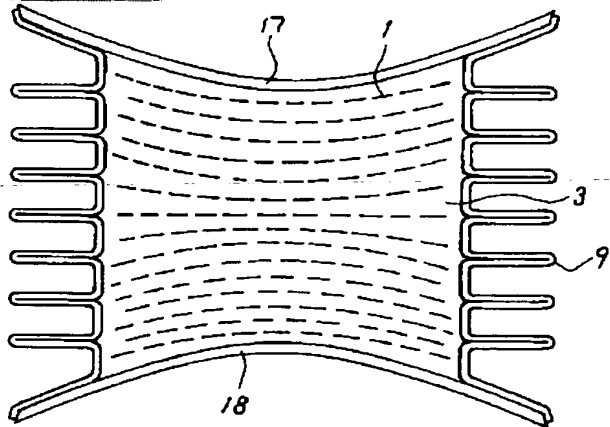
[Drawing 10]



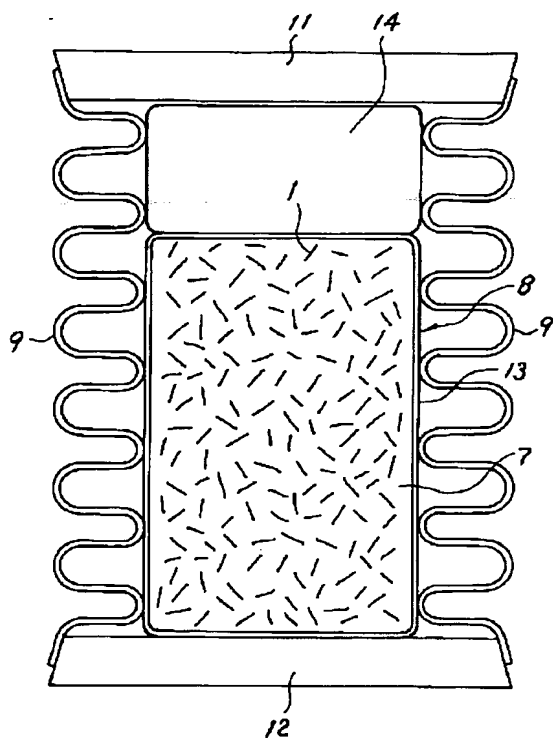
[Drawing 7]



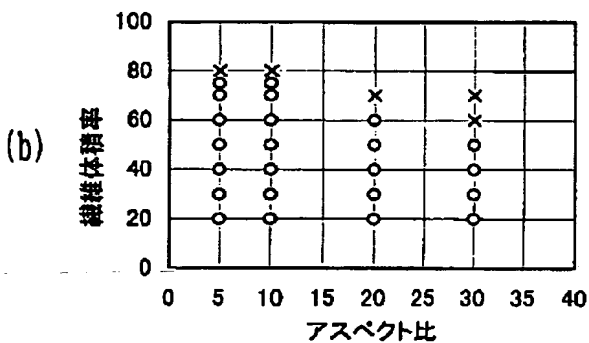
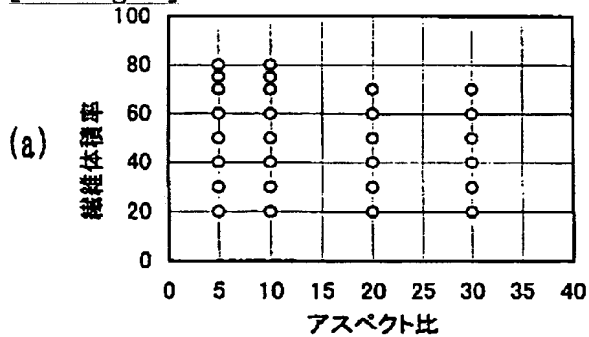
[Drawing 8]



[Drawing 9]

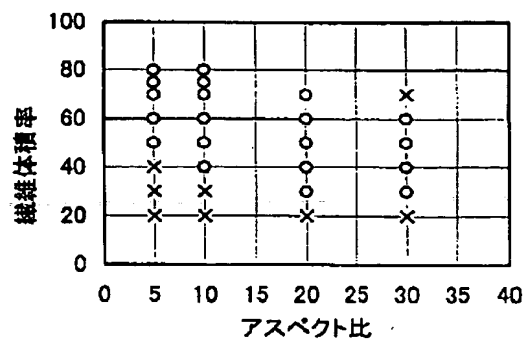


[Drawing 12]

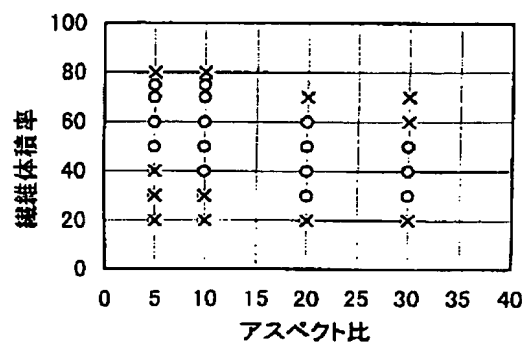


[Drawing 13]

(c)



(d)



[Translation done.]

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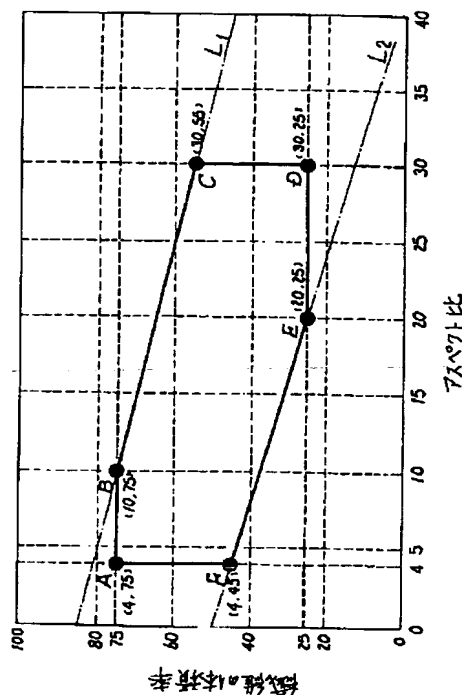
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(54) 【発明の名称】 複合材料及びその製造方法

(57) 【要約】

【課題】 炭素及び／又は黒鉛質からなる繊維によって強化され、面方向に低熱膨張係数を有する複合材料及びその製造方法を提供する。

【解決手段】 炭素及び／又は黒鉛質の繊維と、銅もしくは銅合金からなるマトリックスにより構成され、前記炭素及び／又は黒鉛質の繊維は、その長さが40 μm を越えるもので、アスペクト比及び体積率が図1に示すA(4, 75)、B(10, 75)、C(30, 55)、D(30, 25)、E(20, 25)、F(4, 45)の点に囲まれた範囲であり、かつ2次元面方向にランダムに配向させたことを特徴とする炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料であり、面方向に低熱膨張係数を持ち半導体装置用基板の材料として好適なものである。



【特許請求の範囲】

【請求項1】 炭素及び／又は黒鉛質の繊維と、銅もしくは銅合金からなるマトリックスにより構成され、前記炭素及び／又は黒鉛質の繊維は、その長さが40 μ mを越えるもので、アスペクト比及び体積率が図1に示すA(4, 75)、B(10, 75)、C(30, 55)、D(30, 25)、E(20, 25)、F(4, 45)の点に囲まれた範囲内であり、かつ2次元面方向にランダムに配向させたことを特徴とする炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料。

【請求項2】 複合材料が、半導体装置用基板として用いられることを特徴とする請求項1に記載の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料。

【請求項3】 炭素及び／又は黒鉛質からなる繊維とマトリックスの粉末からなる混合物の圧縮成型物を蛇腹状側壁の金属製カプセルに装填して脱気後密封し、熱間静水圧加圧により、前記圧縮成型物を密封した金属製カプセルを1軸方向に収縮させることを特徴とする請求項1又は2に記載の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法。

【請求項4】 金属製カプセルの蓋部分の肉厚が、蛇腹状金属製カプセルの側壁部の肉厚の2倍以上であることを特徴とする請求項3に記載の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法。

【請求項5】 圧縮成型物を蛇腹状側壁の金属製カプセルに装填し、スペーサーを入れ脱気後密封することを特徴とする請求項3に記載の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法。

【請求項6】 炭素及び／又は黒鉛質からなる繊維とマトリックスの粉末からなる混合物の圧縮成型物が、銅の薄板もしくは箔で包まれていることを特徴とする請求項3乃至5のいずれかに記載の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料並びその製造方法に係り、特に面方向に低熱膨張係数を持ち半導体装置用基板の材料として好適な炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料並びその製造方法に関するものである。

【0002】

【従来の技術】セラミック等からなる強化材と金属を複合化し、双方の利点を有する様々な材料が開発され広く

実用化されている。そのうちのひとつとして、炭素もしくは黒鉛質繊維と銅の複合材が挙げられる。この複合材料は、炭素の持つ潤滑性と銅の持つ高電気伝導性を活かした集電ブラシや、炭素もしくは黒鉛質繊維の持つ低熱膨張の特性と、銅の持つ高熱伝導性を活かした低熱膨張高熱伝導性の半導体装置用基板などに用いられている。

【0003】半導体装置用基板としてこのような複合材料を使用する際、基板の少なくとも面方向の熱膨張係数を低くする必要があり、そのために面方向に熱膨張が低くなるように人為的に炭素繊維を配置したものが知られている（従来例1、特公昭59-16406号公報、特公昭58-16615号公報）。

【0004】また、炭素繊維のアスペクト比が200以上の比較的長い繊維を混合して固化し、等方的に配向させたものが知られている（従来例2、特公昭61-30013号公報）。また、40 μ m以下という短い炭素繊維をマトリックスである銅もしくはA1の粉末と混合し加圧焼結して得られた半導体装置用放熱部材が提案されている（従来例3、特開平9-64254号公報）。また、A1もしくはA1合金の母相に炭素繊維を2次元方向に無秩序に配向させたものが提案されている（従来例4、特開平4-147654号公報）。

【0005】また、複合材料の製法としては、従来より粉末冶金法が一般的で、金属粉末と炭素等の複合材料の製造にホットプレス法やHIP法がしばしば使われ、とりわけHIP法は、量産性に優れた方法といえる。HIP法で複合材料を製作する場合、混合物の圧縮成型体を金属製カプセルに入れ脱気した後密封し、高温高圧処理することにより複合材料の製作がなされるものである。このHIP法による複合材料の製作で大きな変形が行われた場合、金属製カプセルの胴体部や蓋の中心部が優先的に変形し、金属製カプセルの圧縮成型体に不均一な変形が生ずる。このようなHIP処理時におけるカプセルの不均一な変形を防止することに関し、A1粉末と強化材粉末との混合体の成形において、カプセルの側壁部を蛇腹体とすることが知られている（従来例5、特許第2535401号公報）。

【0006】

【発明が解決しようとする課題】しかしながら、上記従来例1では、長繊維を人為的に配置するのは製作に手間がかかり経済的とはいえない難いものであり、またこの長繊維を配置したものでは、Ag鋳付けなどによる高温の熱履歴を加えると局部的に極めて大きな内部応力が発生し、マトリックスがその内部応力に耐えられず材料が破損もしくは変形が生じる危険が大きいという問題があった。

【0007】上記従来例2では、繊維長がアスペクト比で200以上と比較的長い繊維を使用しており、ホットプレス法を用いると繊維を十分に面方向に配向させることはできず、且つ繊維が曲がったまま固化されやすくな

るため材料中に極めて大きな内部応力が蓄積されることとなり、上記従来例1と同様に熱履歴による材料の破損、変形の危険が大きく十分な信頼性があるとは言えないものである。

【0008】また、上記従来例3では、40 μ m以下という短い繊維を用いているが、これでは各繊維の熱膨張係数低下効果が著しく低くなり所望の熱膨張係数を得るのに極めて多量のカーボン繊維を必要とし、この場合高い熱伝導率を得るには黒鉛化度の高い高級な繊維を用いる必要があり、高コストであるという問題がある。また、上記従来例4では、A1の母相に炭素繊維を2次元方向に無秩序に配向させたものであるが、半導体装置用に適用した場合、十分な信頼性があるものとは言えないものである。

【0009】また、上記従来例5では、HIP処理のカプセル側壁部を蛇腹体することが示されているが、これはA1粉末と強化材粉末との混合体の成形についてのものです。カプセルの不均一変形を阻止すると同時に、銅もしくは銅合金からなるマトリックス中の炭素及び／又は黒鉛質繊維の配向を揃えるという作用については開示されていないものである。

【0010】本発明は、炭素及び／又は黒鉛質からなる繊維によって強化され、面方向に低熱膨張係数を有し、特に半導体装置用基板の材料として好適な炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料並びにその製造方法を提供するものである。

【0011】

【課題を解決するための手段】本発明は、炭素及び／又は黒鉛質の繊維と、銅もしくは銅合金からなるマトリックスにより構成され、前記炭素及び／又は黒鉛質の繊維は、その長さが40 μ mを越えるもので、アスペクト比及び体積率が図1に示すA(4, 75)、B(10, 75)、C(30, 55)、D(30, 25)、E(20, 25)、F(4, 45)の点に囲まれた範囲内であり、かつ2次元面方向にランダムに配向させたことを特徴とする炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料である。

【0012】本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料は、そのアスペクト比及び体積率が、好ましくは、図2のH(5, 70)、I(15, 60)、J(20, 60)、K(20, 40)、L(10, 40)、M(5, 50)の点に囲まれた範囲内であることを特徴とするものである。なお、本発明において、体積率とは、(炭素及び／又は黒鉛質の繊維の体積) / (炭素及び／又は黒鉛質の繊維の体積 + マトリックスの銅もしくは銅合金の体積) である。

【0013】また本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料は、マトリックスが銅もしくは、さらにMo、W、C

r、Ag、セラミック粒子の1種又は2種以上を含む銅合金であることを特徴とするものである。また本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料は、半導体装置用基板として用いられることを特徴とするものである。

【0014】また本発明は、炭素及び／又は黒鉛質からなる繊維とマトリックスの粉末からなる混合物の圧縮成型物を蛇腹状側壁の金属製カプセルに装填して脱気後密封し、熱間静水圧加圧により、前記圧縮成型物を密封した金属製カプセルを1軸方向に収縮させることを特徴とする炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法である。

【0015】また本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法は、金属製カプセルの蓋部分の肉厚が、蛇腹状金属製カプセルの側壁部の肉厚の2倍以上であることを特徴とするものである。また本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法は、圧縮成型物を蛇腹状側壁の金属製カプセルに装填し、スペーサーを入れ脱気後密封することを特徴とするものである。

【0016】また本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法は、炭素及び／又は黒鉛質からなる繊維とマトリックスの粉末からなる混合物の圧縮成型物が、銅の薄板もしくは箔で包まれていることを特徴とするものである。

【0017】さらに、本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造方法は、熱間静水圧加圧が、800℃以上で、銅もしくは銅合金の融点以下の温度で行われることを特徴とするものである。

【0018】

【作用】本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料によれば、炭素及び／又は黒鉛質からなる繊維によって強化され、面方向に低熱膨張係数を有し、また焼結固化による内部応力が小さく、熱履歴による変形、破損の危険がないものである。また、本発明は、炭素及び／又は黒鉛質の繊維が2次元面方向にランダムに配向していることで、収縮軸に垂直な面方向の熱膨張係数は低い値をとることができ、高熱伝導性の黒鉛化率の高い黒鉛質繊維を用いることにより、この方向に高熱伝導とすることができるので、この面に対し平行な向きに材料を切り出せば半導体装置用基板として好適な製品を得ることができる。

【0019】また、本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料の製造は、熱間静水圧加圧(HIP法)で蛇腹状側壁の金属製カプセルを用い、1軸方向に収縮させるので、炭素及び／又は黒鉛質からなる繊維を収縮方向に対して垂

直な方向に配向させることができるものである。

【0020】本発明の複合材料は、マトリックスとして銅もしくは銅合金を用いるので、熱伝導性が良好である。またマトリックスがA1の複合材料の場合は、炭素、黒鉛の繊維とA1との界面に、Al₄C₃等の有害な炭化物が形成され、温度サイクル時に界面で剥離しやすく、炭素、黒鉛繊維による熱膨脹低下効果が十分に得られず、信頼性の点で問題があるが、銅もしくは銅合金のマトリックスではかかる問題がない。さらに、パッケージと放熱基板とのAgろう付けが可能な耐熱性を有し、セラミックパッケージに対する放熱基板としても有用なものである。

【0021】本発明の炭素及び／又は黒鉛質繊維のアスペクト比及び体積率の限定理由を説明する。図1は、横軸にアスペクト比、縦軸に炭素及び／又は黒鉛質からなる繊維の体積率を示し、炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料のアスペクト比と体積率の関係を示す図である。本発明の炭素及び／又は黒鉛質からなる繊維のアスペクト比及び体積率は、図1に示すA(4, 75)、B(10, 75)、C(30, 55)、D(30, 25)、E(20, 25)、F(4, 45)の点に囲まれた範囲内である。

【0022】アスペクト比とは、繊維長／繊維径である。本発明のアスペクト比の限定理由は、アスペクト比が4より小さい炭素及び／又は黒鉛質からなる繊維は、作製が困難でありコスト高になるのでアスペクト比4以上のものを用いた。またアスペクト比が30を越えると複合材料を焼結固化した際に蓄積される内部応力が大きくなりすぎ、熱履歴による変形、破損の危険が大きいの

で30以下に限定した。

【0023】体積率の限定理由は、炭素及び／又は黒鉛質繊維の体積率が、75%を越えると焼結固化が困難となり、たとえ焼結できても脆くなり、複合材料としての特性が得られず、特に半導体装置用基板として要求される特性を満たすことはできない。また体積率が、25%より小さいと熱膨脹係数が大きくなりすぎて適当ではないので体積率は25%以上75%以下に限定される。

【0024】さらに、これらの条件を満たしていても、図1における線L1(一点鎖線)より上部の領域では、焼結固化した際の内部応力が大きくなるため、熱履歴により変形、破壊を生じるため適当でない。また線L2(一点鎖線)よりより下部の領域では熱膨脹係数を小さくすることはできない。特に半導体装置用基板として適当な熱膨脹係数の値まで下げることはできない。このような理由により図1に示すA(4, 75)、B(10, 75)、C(30, 55)、D(30, 25)、E(20, 25)、F(4, 45)の点に囲まれた領域に炭素及び／又は黒鉛質繊維のアスペクト比及び繊維の体積率を特定した。特に好ましいのは、アスペクト比と体積率

の関係を示す図2のH(5, 70)、I(15, 60)、J(20, 60)、K(20, 40)、L(10, 40)、M(5, 50)の点に囲まれた領域である。

【0025】また、2次元面方向に繊維を無秩序に配向させるのは、それにより内部応力の集中が問題になるような長い繊維を用いなくても、アスペクト比と体積率で規定される最小の繊維量で所定の低熱膨脹が得られるからである。図3は、本発明の複合材料の概要を示す図

で、複合材料(2)は、銅もしくは銅合金からなるマトリックス(3)に、炭素及び／又は黒鉛質繊維(1)が2次元面方向にランダムに配向されているものである。

【0026】本発明の複合材料は、面方向で低い熱膨脹係数が得られるものである。半導体装置用基板として適用するときは、半導体装置の形態にもよるが熱膨脹係数は5~11ppm/°Cが適当であり、その中でも7.0~9.0ppm/°Cが好ましいものであり、本発明の複合材料の面方向の低熱膨脹係数はかかる要求に対応できるものである。

【0027】

【発明の実施の形態】本発明の炭素及び／又は黒鉛質繊維のアスペクト比及び体積率について上述したが、炭素及び／又は黒鉛質の繊維の径は、5~20μmのものが用いられ、好ましくは8~12μmのものである。その長さが40μmを越えるものである。なお、繊維の平均長さの上限はそのアスペクト比(繊維長／繊維径)で定まる。

【0028】また、本発明のマトリックスは銅もしくは銅合金である。マトリックスとしては銅のみでもよいが、銅もしくは銅に、さらにMo、W、Cr、Ag、セラミック粒子の1種又は2種以上を添加した銅合金を用いてもよい。用途に応じて、例えば強度の上昇を意図してMo、W、Cr等の高融点金属の1種又は2種以上を添加する。またセラミック粒子を添加する。また耐熱性を向上させるためにAgなどを添加してもよい。

【0029】本発明の複合材料の製造方法は、炭素もしくは黒鉛質からなる繊維を、マトリックスをなす銅を主とする粉末と共にボールミル等の手法で混合する。また、マトリックスとして、CuにMo、W、Cr、Agの粉末の1種又は2種以上を、またはセラミック粒子を添加し、ボールミル等の手法で混合して得た原料混合物を圧粉型に入れプレスして混合物の圧縮成型物を得る。

【0030】炭素及び／又は黒鉛質からなる繊維とマトリックスの粉末からなる混合物の圧縮成型物をHIP(熱間静水圧加圧)法を用いて焼結固化させる工程について図面を参照して説明する。図4~図6は、本発明の圧縮成型物をHIPで焼結固化させる工程を示すもので、まず図4に示すように、炭素及び／又は黒鉛質の繊維(1)とマトリックスの粉末(7)との圧縮成型物(8)を側壁を蛇腹体(9)で構成される金属製カブセ

ルに装填し、蓋(11)(12)と蛇腹体(9)を真空中で電子ビーム溶接するなどして真空密封する。

【0031】側壁を蛇腹体(9)で構成される金属製カプセルの材料としては、主に、ステンレス鋼が用いられる。ステンレス鋼の中でもSUS304が溶接性の点から望ましい。また蛇腹体(9)と、蓋(11)(12)は同一の材料で構成することが好ましい。また、側壁を蛇腹体で構成される金属製カプセルは、円筒状、角筒状いずれでもよい。加圧の均一性という点では円筒状が好ましく、ピレットから所定の部材を得る際の歩留まりという点では角筒状が好ましい。また、金属製カプセル側壁の蛇腹体は、円弧状のものを図示したが、これに限るものではなく、三角状の蛇腹でもよい。

【0032】また、炭素もしくは黒鉛質からなる繊維の体積率が大きくなると、圧縮成型物の保形性が失われるので、銅薄板もしくは箔で包むことが好ましい。例えば、予め圧粉用型の内面に沿って銅箔(13)を敷き詰め、その中に炭素もしくは黒鉛質からなる繊維(1)とマトリックス粉体(7)の混合物を充填し圧粉することにより圧粉体の圧縮成型物(8)が銅箔(13)に包まれた形態をとるようにして保形性を与えるようにして、図5に示すように、側壁を蛇腹体(9)で構成される金属製カプセルに装填し、蓋(11)(12)をして真空密封することが好ましい。

【0033】次いで、図6に示すように、側壁が蛇腹体(9)の金属製カプセルに装填され、蓋(11)(12)で真空密封された圧縮成型物(8)をHIP処理する。金属製カプセル側壁の蛇腹体(9)は、その胴体の軸方向に収縮する。収縮が進行すると、金属製カプセル胴体の軸に垂直な面方向の強度は増すが、さらに軸方向に収縮が進行する。この結果軸に垂直な面内ではどこでも同じ加圧収縮が起こるので、この面内では炭素もしくは黒鉛質からなる繊維(1)の体積比、配向は同じとなる。このように加圧し1軸方向に収縮させることにより、炭素もしくは黒鉛質からなる繊維(1)は収縮軸方向に垂直な面内に配向し、マトリックス(3)に、炭素及び／又は黒鉛質繊維(1)が2次元面方向にランダムに配向されているものとなり、本発明の複合材料が容易に且つ高い歩留まりで得ることができる。

【0034】本発明の複合材は、加圧し1軸方向に収縮させることにより、マトリックス(3)に、炭素及び／又は黒鉛質繊維(1)が2次元面方向にランダムに配向されているものであるが、これについて図7(a)

(b)で検討した。図7(a)に示すように、金属製カプセル(23)に炭素、黒鉛質の繊維(1)とマトリックスの粉末(20)との圧縮成型物を装填し、蓋(21)(22)で真空密封する。これにHIP処理を行ったところ、図7(b)に示すように、金属製カプセル(23)、及び蓋(21)(22)の中心部が優先的に変形し、その変形状況に応じて、繊維(1)の配向が全

体的に大きくばらついた。

【0035】そこで、本発明の複合材料の製造では、蛇腹状側壁の金属製カプセルを用いて、熱間静水圧加圧(HIP法)で1軸方向に収縮させ、炭素及び／又は黒鉛質からなる繊維を収縮方向に対して垂直な方向に配向させるものである。また、蛇腹状側壁の金属製カプセルを用いても、カプセル変形の不均一が発生することもある。例えば図8に示すように、圧縮成型物のHIP処理により、側壁が蛇腹体(9)の金属製カプセルに装填され、蓋(17)(18)で真空密封された圧縮成型物は、側壁が蛇腹体(9)からなる胴体の軸方向に収縮するが、蓋(17)(18)の中心部が変形し、繊維(1)の配向が全体的にばらつくことがある。このような、不均一変形の発生に対応するために、金属製カプセルの蓋を、側壁をなす蛇腹体の肉厚の2倍以上とすることにより、蓋よりも側壁をなす蛇腹体を優先的に変形させ、十分に充填密度を上げる。このようにすると、十分に充填密度が上がるまで蓋に大きな圧力がかかるのが防止できるので蓋のへこみを抑制できる。

【0036】金属製カプセルの蓋を側壁蛇腹体の肉厚の2倍以上の厚さにする代わりにスペーサーを用いてもよい。例えば、図9に示すように、炭素及び／又は黒鉛質の繊維(1)とマトリックスの粉末(7)との銅箔(13)で包まれた圧縮成型物(8)を側壁蛇腹体(9)の金属製カプセルに装填し、スペーサー(14)を入れ、蓋(11)(12)をして真空密封する。スペーサーの材質としては無酸素銅、黒鉛が好ましい。これをHIP処理すると図10に示すように、金属製カプセルの側壁蛇腹体(9)は1軸方向に収縮し、マトリックス(3)に、炭素及び／又は黒鉛質繊維(1)が2次元面方向にランダムに配向されたものとなる。また、図9では、金属製カプセルに挿入されるスペーサーを圧縮成型物の上に配置しているが、圧縮成型物の上、下に配置してもよい。

【0037】本発明の炭素及び／又は黒鉛質の繊維と銅もしくは銅合金のマトリックスよりなる複合材料を半導体装置用基板として用いる場合について図11を参照して説明する。本発明の複合材料は、炭素及び／又は黒鉛質の繊維が2次元面方向にランダムに配向されているもので、このような複合材料を半導体基板の面方向に繊維が配向しているようにして半導体装置用に適用する。

【0038】具体的には図11に示すように、本発明の複合材料を基板の面方向に繊維が配向しているように切り出し所定の形状に加工し、半導体装置用基板(4)として用いる。半導体装置用基板(4)には、必要に応じてNi, Au, Pd, などからなるめっき層を形成し、その上に半導体素子(6)を搭載する。また半導体装置用基板(4)は、アルミナやコパールなどからなるパッケージの外囲器(5)と接合する。

【0039】このように本発明の複合材料を半導体装置

用基板として用いるが、アスペクト比、繊維体積率を制御することにより、各種半導体パッケージ材料と接合するのに好適な熱膨張係数とすることができる。それ故、該基板とアルミナやコパールなどからなるパッケージの外囲器と接合する際の熱膨張差に起因する反り等を抑制でき、またガラスセラミックなどからなる端子と反り等発生することなく接合できる。また発熱する半導体チップとの間に発生する熱応力が緩和できるため、信頼性の高い半導体装置が得られる。

【0040】

【実施例1】本発明の第1の実施例を図5、図6及び表1、表2を参照して説明する。まず、繊維径10 μ m、平均繊維長200 μ mの黒鉛質繊維、及び平均径20 μ mの銅粉を用い、黒鉛質の繊維の体積率が50%になるように、所定重量の繊維、銅粉を秤量して配合し、ボールミルにて混合し、これを予め内壁に沿って厚さ35 μ mの銅箔を敷き詰めた金型に入れ、1t/cm²の圧力で乾式プレスして圧縮成型物を形成した。この圧縮成型物の充填率（圧縮成型物の体積の内、繊維と銅粉の混合物が占める割合）は40%程度であった。

【0041】次いで、上述のように形成した圧縮成型物を図5に示すように金属製カプセルに装填する。銅箔（13）に包まれた黒鉛質繊維（1）とマトリックスとなる銅粉（7）の圧縮成型物（8）を、内径65mm、肉厚0.6mm、蛇腹のピッチ10.0mmで、材質がSUS304からなる蛇腹状側壁（9）を有する金属製カプセルの中に入れ、これの端面に厚さ2mmの蓋（11）（12）を真空中で電子ビーム溶接することにより、真空封入した。

【0042】次いで、1500気圧、1000℃2hの*30

密度(g/cm³)

中心からの距離(cm)	0	1	2	3
実施例	5.55	5.55	5.55	5.55
比較例	5.55	5.55	5.53	5.49

【表2】

熱伝導率(W/mK)

中心からの距離(cm)	0	1	2	3
実施例	390	387	392	389
比較例	391	381	365	339

【0045】表1から明らかなように、実施例1ではサンプル採取位置が、中心からの距離が0cm、1cm、2cm、3cmのいずれも、密度が5.55（g/cm³）で一定である。これに対し、比較例では密度にばらつきが生じている。また、表2に示す熱伝導率も、実施例1ではサンプル採取位置によらず、ほぼ一定である。これに対して比較例は、ばらつきが生じている。

【0046】これら表1、表2の結果より、実施例1ではサンプル採取位置によらず、密度が一定であることから繊維とマトリックスの銅の体積比は一定といえる。ま

*条件でHIP処理した。図6に示すように、HIP処理により金属製カプセルの蛇腹状側壁（9）は、その円筒の軸方向に沿って一様に潰れて、圧縮成型物は1軸方向に収縮された。圧縮成型物（8）の高さ約60mmのものが、1軸方向に収縮され、高さ約25mmの繊維強化複合材料が得られた。また、黒鉛質繊維（1）は銅のマトリックス（3）に2次元面方向にランダムに配向されていた。

【0043】比較例として、図7（a）（b）に示すようにHIP処理を行ってみた。上記実施例1と同様に、圧縮成型物を形成し、黒鉛質繊維（1）とマトリックスとなる銅粉（20）の圧縮成型物を、内径65mm、肉厚0.6mmの円筒状の金属カプセル（23）に入れ、蓋（21）（22）で同様に真空密封した。これを1500気圧、1000℃2hの条件でHIP処理した。処理終了後、これらをHIP装置から取り出したところ、図7（b）に示すように金属カプセル（23）、及び蓋（21）（22）の中心部分が大きくへこみびつな形となり、その変形状況に応じて、繊維（1）の配向が全体的に大きくばらついていた。

【0044】上記実施例1及び比較例の複合材料について中心から1cmごとに直径9mm厚さ4mmのサンプルを採取した。なお、サンプルは、HIP処理の金属製カプセルの円筒軸方向と垂直になるように採取したものである。表1は、実施例1及び比較例のサンプルの密度（g/cm³）を測定したものであり、表2は、レーザーフラッシュ法にて熱伝導率（W/mK）を測定したものである。

【表1】

た、熱伝導率が一定であることから、サンプルの採取位置によらず、圧縮軸と垂直な向きではどこでも繊維の配向状態が同じであると言える。それに対し、比較例では、密度にもばらつきが生じ、熱伝導率にはさらに多くのばらつきが生じた。このことから図7（b）に示すように、カプセルのいびつな変形により繊維の体積比、配向がサンプル採取位置により大きく異なっていることが判る。

【0047】この実施例1のサンプルを酸を用いて溶解し、平均のアスペクト比を求めたところ、10.2であ

った。なお、原料の黒鉛質繊維は、繊維径 $10\mu\text{m}$ 、平均繊維長 $200\mu\text{m}$ のものであるが、ボールミルでの混合、乾式プレスによる圧縮成型、またはHIP等により得られた複合材料の平均アスペクト比は、10.2のものであった。

【0048】

【実施例2】第2の実施例を図12(a)(b)及び図13(c)(d)を参照して説明する。図12(a)(b)及び図13(c)(d)において、横軸はアスペクト比、縦軸は炭素及び／又は黒鉛質からなる繊維体積率である。炭素質の繊維として繊維径 $10\mu\text{m}$ 、Cu-20vol%M合金をマトリックスとして実施例1と同様の製法で、様々のアスペクト比、様々の体積率を持つ複合材料を作製した。これを還元雰囲気中 850°C で2h熱処理し、その変形挙動を調査した。またその熱膨張係数を測定し適当な値(5~11ppm)であるかを調査した。これらを総合して各複合材料が半導体装置用基板として適当な特性を備えているか調べた。

【0049】図12(a)は、作製した複合材料を○印で示したものである。図12(b)は、熱処理による変形挙動を示したもので、○印は変形しないもの、もしくは変形が軽微なものであり、×印は著しく変形したものの、もしくは破壊したものである。図13(c)は、熱膨張による変形挙動を示したもので、○印は適当な値(5~11ppm)のもの、×印は高すぎるか、低すぎるものである。図13(d)は、総合評価結果を示したもので、○印は半導体装置用基板として適するもの、×印は半導体装置用基板として不適なものである。図12(a)(b)及び図13(c)(d)に示す結果から明らかのように、本発明で特定されている範囲内のアスペクト比及び体積率を持つ場合のみ好適な特性を併せ持つことが判る。

【0050】

【実施例3】第3の実施例を図11を参照して説明する。繊維径 $10\mu\text{m}$ 、平均繊維長 $200\mu\text{m}$ の黒鉛質の繊維と平均径 $10\mu\text{m}$ の銅粉を、黒鉛質繊維の体積率が60%になるように配合し、実施例1と同様に複合材料を作製した。実施例1と同様にアスペクト比を求めたところ7.5であった。この複合材料を黒鉛質繊維の配向面が面方向となるように切り出し基板とし、図11に示すように、半導体装置用基板(4)にアルミナからなる外囲器(5)をAg糊付けした。この際、基板(4)に*

*反り等は見られなかった。さらに、これに半導体素子(6)を取り付け、 $-40^\circ\text{C} \rightarrow 125^\circ\text{C}$ の温度サイクルを500サイクルかけた。その結果、半導体素子(6)の基板(4)からの剥離は認められなかった。

【0051】

【実施例4】第4の実施例を、図5~図10及び表3、表4を参照して説明する。上述した実施例1と同様に原料混合物を混合して圧縮成型物を形成し、実施例1と同様のHIP処理をした。1つは(表3、表4の「蓋厚み2.0mm」)、図5に示すように、圧縮成型物を側壁が内径 150mm 、肉厚 1.0mm 、ピッチ 15mm の蛇腹体筒(9)からなる金属製カプセルに入れ、厚みが 2.0mm の蓋(11)(12)をして真空封入した。

【0052】もう1つは(表3、表4の「蓋厚み $0.6\text{mm} + \text{スペーサ}$ 」)、図9に示すように、圧縮成型物を側壁が内径 150mm 、肉厚 1.0mm 、ピッチ 15mm の蛇腹体筒(9)からなる金属製カプセルに装填し、無酸素銅からなる厚さ 10mm のスペーサ(14)を入れ、厚みが 0.6mm の蓋(11)(12)をして真空封入した。さらに(表3、表4の「蓋厚み 0.6mm 」)、図8に示すように、圧縮成型物を側壁が内径 150mm 、肉厚 1.0mm 、ピッチ 15mm の蛇腹体筒(9)からなる金属製カプセルに入れ、厚みが 0.6mm の蓋(17)(18)をして真空封入した。なお、いずれの場合も、蛇腹体の金属製カプセル及び蓋の材質はSUS304を用いた。

【0053】HIP処理を行った結果、図5に示す「蓋厚み 2.0mm 」の場合は、図6に示すように蓋(11)(12)は互いに平行であり、へこみ等は見られず、圧縮成型物は1軸方向に収縮した。また、図9に示す「蓋厚み $0.6\text{mm} + \text{スペーサ}$ 」の場合は、蓋の厚みは 0.6mm であるが、厚さ 10mm のスペーサを入れたので、図10に示すように、へこみ等は発生しなかった。これに対して、「蓋厚み 0.6mm 」では、図8に示すように、蓋(17)(18)の中心部が変形した。その変形は蓋(17)(18)の中央部が周辺部に対し約 3mm 陥没していた。

【0054】これらの材料について、上記実施例1と同様にサンプルを採取し、密度及び熱伝導率を測定した。表3は密度(g/cm^3)、表4は熱伝導率(W/mK)を示したものである。

【表3】

密度(g/cm^3)				
中心からの距離(cm)	0	1	2	3
蓋厚み 2.0mm	5.55	5.55	5.55	5.55
蓋厚み $0.6\text{mm} + \text{スペーサ}$	5.55	5.55	5.55	5.55
蓋厚み 0.6mm	5.55	5.55	5.54	5.52

【表4】

熱伝導率(W/mK)

中心からの距離(cm)	0	1	2	3
蓋厚み2.0mm	390	387	392	389
蓋厚み0.6mm + スペース	392	389	387	390
蓋厚み0.6mm	391	388	378	365

【0055】表3、表4の結果より、「蓋厚み2.0mm」、「蓋厚み0.6mm + スペース」はサンプル採取位置によらず、密度が一定であることから繊維とマトリックスの銅の体積比は一定であり、圧縮軸と垂直な向きではどこでも繊維の配向状態が同じであり、熱伝導率が一定であると言える。これらに対し、「蓋厚み0.6mm」のへこみが発生したものでは、密度にもばらつきが生じ、熱伝導率にもばらつきが生じた。

【0056】

【発明の効果】以上説明したように、本発明の複合材料及びその製造方法によれば、炭素及び／又は黒鉛質からなる繊維によって強化され、面方向に低熱膨張係数を有し、また焼結固化による内部応力が小さく、熱履歴による変形、破損の危険がないという効果を奏するものである。

【図面の簡単な説明】

【図1】本発明のアスペクト比及び体積率の関係を示す図

【図2】本発明のアスペクト比及び体積率の関係を示す図

【図3】本発明の複合材料の概要を示す図

【図4】本発明の実施態様及び実施例を説明する図

【図5】本発明の実施態様及び実施例を説明する図

*【図6】本発明の実施態様及び実施例を説明する図

【図7】比較例を示す図

【図8】比較例を示す図

【図9】本発明の実施態様及び実施例を説明する図

10 【図10】本発明の実施態様及び実施例を説明する図

【図11】本発明の実施態様及び実施例を説明する図

【図12】本発明の実施例のアスペクト比及び体積率の関係を示す図

【図13】本発明の実施例のアスペクト比及び体積率の関係を示す図

【符号の説明】

1 炭素もしくは黒鉛質からなる繊維

2 複合材料

3 マトリックス

20 4 半導体装置用基板

5 半導体装置の外囲器

6 半導体素子

7 マトリックスの粉末

8 圧縮成型物

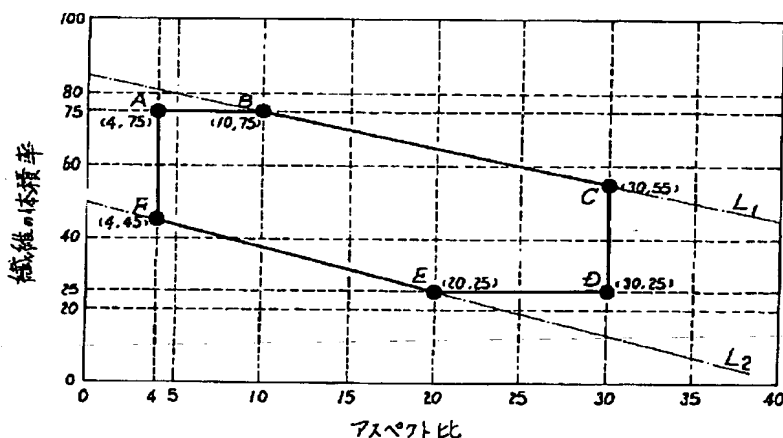
9 蛇腹体

11、12 蓋

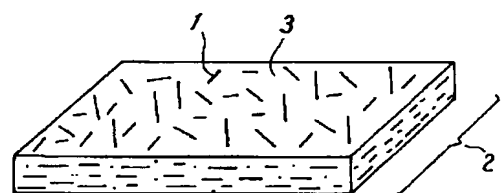
13 銅箔

* 14 スペース

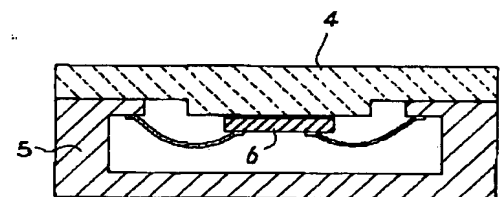
【図1】



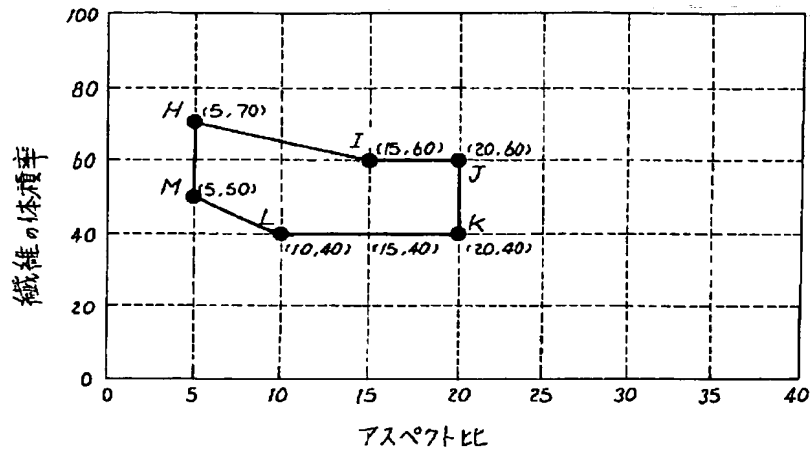
【図3】



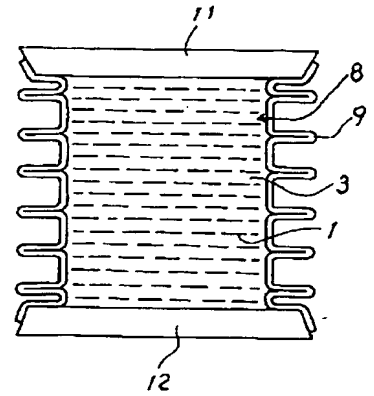
【図11】



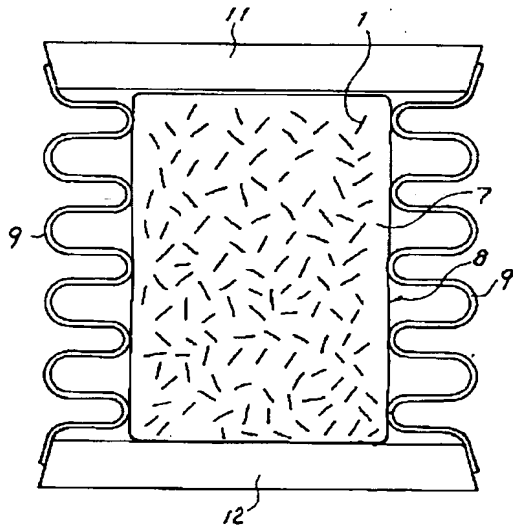
【図2】



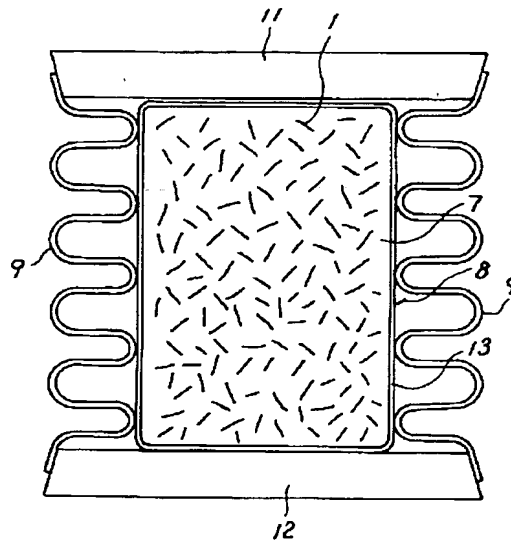
【図6】



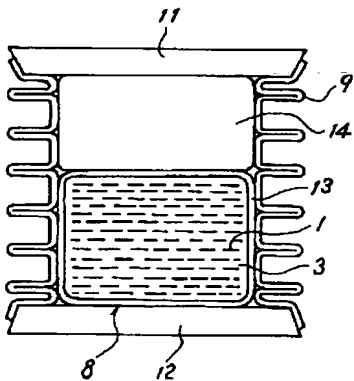
【図4】



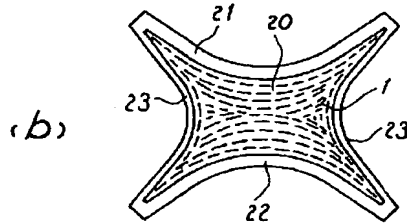
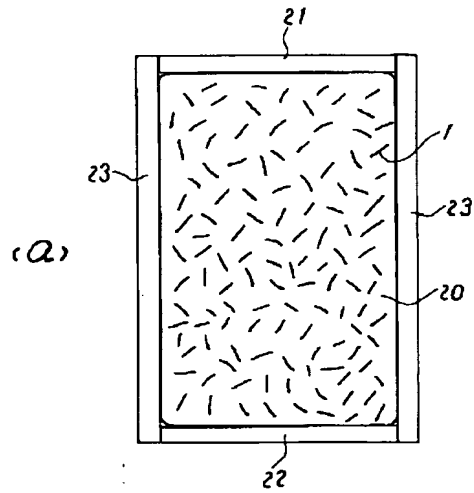
【図5】



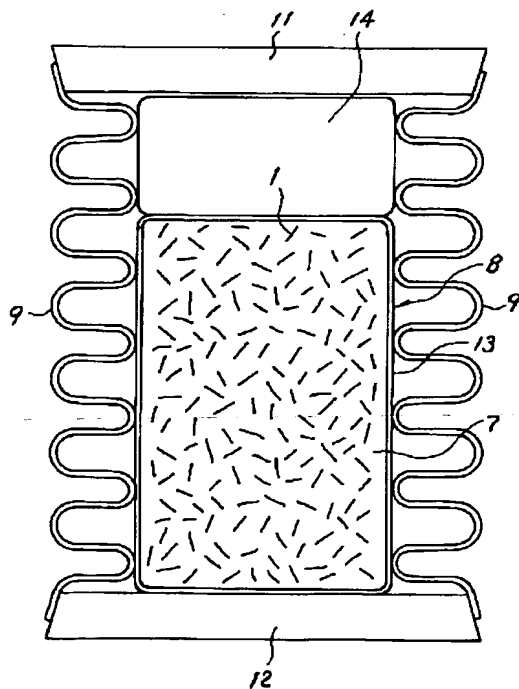
【図10】



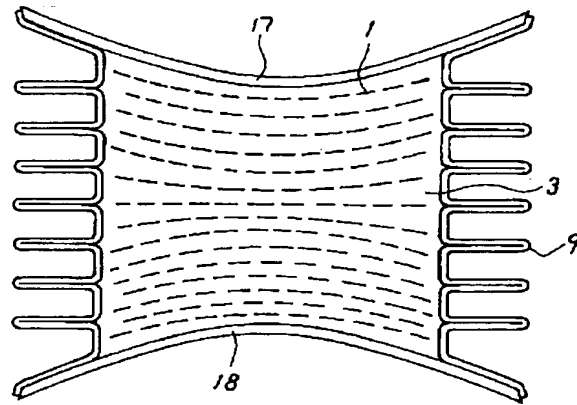
【図7】



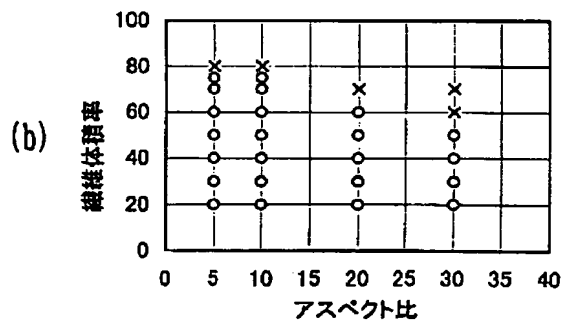
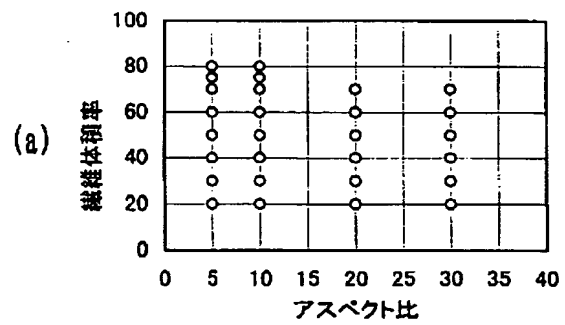
【図9】



【図8】



【図12】



〔図13〕

